77

levels. The raw odour of the ethanol may be quite difficult to mask and the residual odour on the hair may need careful control.

2.5.1.6 Other additives Preservatives are unnecessary in ethanol-based products such as hairsprays. Many other additives, such as vitamins, proteins, amino acids and herbal extracts, are used. Most of these are present in very small amounts and cannot seriously be expected to dramatically affect product performance. Ultraviolet filters have become a fashionable addition and some work has demonstrated that they may be of value in leave-on styling products [88, 93, 94], although some of the studies have used unrealistically high levels of UV filters.

There seems to be little doubt that hair can be damaged appreciably by ultraviolet light (and other environmental influences). The nature and extent of this damage, which includes photo-oxidation, loss of mechanical strength, increased alkaline solubility and colour changes are examined by Dubrief [94].

: Setting lotions

e fundamental difference between hairsprays and setting lotions is that latter are intended for application to wet hair. Water-soluble polymers therefore used and the alcohol content, tends to be lower. Completely based products are possible since drying time is not so critical. Use

22	Formulations	for	a	setting/blow-dry	lotion
----	--------------	-----	---	------------------	--------

	<u> </u>				
mol.B96	40	40	18	30	
glaternium-11, 50%	2			50	_
rogenated tallow dimethyl benzyl	-				_
iliterium chloride, 75%	0,2				
un-polystyrene sulphonate, 50%	_	_	2.2		
hyrrolidone/vinyl acetate copolymer,			2.2		
			_	1.2	2
agetate/crotonic acid copolymer	_	1.2	_		
lemum-22, 60%		0.4	0.2		
iviyinylimidazolium chloride/vinyl					
fiolidone copolymer 95/5, 40%	_	_		0.5	0.25
OLY1161-9	0.25				0.3
	0.1	0.05	0.15	0.1	0.1
negreta -		0.2		0.1	0.2
opiopy adipate –		0.2	_		
	_		0.5	0.25	
atonium benzoate, 2.5% solution	_	_	0.005		_
onio Anitropropane-1,3-diol			0.03	0.01	
HARRINGFOISOthiazolinone and			2.33	V.VI	
einviscilniazolinone, 1.5%		_			0.05
Inten Water	to 100				0.03
	.5 100				

THIS PAGE BLANK (USPTO)

of acidic polymers with a higher degree of neutralisation is another wide practised option. Some products are marketed specifically as blowed lotions, but are essentially similar. Since the action of these products is instant' in the manner of a hairspray, and since levels of hold are general lower, there is more scope to incorporate conditioning ingredients; a blen based on a cationic polymer plus a hairspray-type resin can give good result A strongly anionic resin, sodium polystyrene sulphonate, has been successfully used since its highly conductive film is effective in reducing static. The can be important during blow-drying, when, towards the end of the process the hair is virtually dry and can exhibit 'fly-away', making styling difficult This problem is not as obvious with other forms of heating devices such a tongs and heated curlers. Typical formulations for a setting/blow-dry lotion are shown in Table 2.22.

2.7 Other styling aids in spray form

Names such as 'spritz', 'spray gel', 'sculpting spray', etc. are usually hairspray' or setting lotion variants of one sort or another. Often a 'wet-look' or glazed effect is desired, and this usually demands a high resin content in a water or water/ethanol base. Since styling of the wet hair is carried out, the product must not dry too quickly and must be compatible with water. Especial care must be taken with the high-gloss products not to reduce gloss by injudicious choice of plasticisers. Likewise, the high solids contents of some formulations may restrict pump-spray choice. Typical formulations for wet-look styling sprays are given in Table 2.23. The products are popular in other physical forms such as gels and creams (see sections 2.8 and 2.9).

A few other spray products, claiming specific properties such as volume enhancement and gloss, are available (see Table 2.24). Almost any hair-styling aid enhances volume by virtue of the deposited polymer, which increases the diameter of the hair shaft, This is much more obvious in the case of products applied to dry hair; 'wet-look' and sculpted styles tend to reduce volume by sticking adjacent hairs together in parallel lines, whereas a more random orientation of the individual hairs gives a more bulky appearance. Over a

Table 2.23 Formulations for wet-look styling sprays (applicable to wet hair)

Deionised water	o wet hair)	
Vinyl caprolactam/vinyl pyrrolidone/dimethylaminoethyl	to 100	
Lauryl pyrrolidone/dimethylaminoethyl methacrylate 20%	6.5	6
2-Bromo-2-nitropro-2	0.3	4
Methyldibromoglutaronitrile (and) phenoxyethanol	0.04	0.3
, i mony children		0.05

THIS PAGE BLANK (USPTO)

50.00-300 SZZ-535

Safety of Permanent Waving

Ammonium thioglycolate now has a long history of safe consumer us Allergic reactions to thioglycolate home permanents are extremely ran if they occur at all. (Storrs 1984) However, glycerol monothioglycolla has been clearly shown to cause sensitization reactions in hairdresse and in some cases their clients (Storrs 1984; Guerra et al. 1992; Matsi naga et al. 1988) and hairdressers occasionally become sensitive ATG as well. (Matsunaga et al. 1988) A study among European Ha dressers found 19% sensitized to GMT and only 4% sensitized to ATC (Frosch et al. 1993) The report of the Cosmetic Ingredient Review (G) of the Cosmetic, Toiletry and Fragrance Association (CTFA) on thiog colates recommends that thioglycolates may be safely used by col sumers at infrequent intervals, but recommends that hairdressers avo skin contact. (Elder 1991) A recent study by Uter et al. (2000) four that the incidence of sensitivity to GMT in German hairdressers in been declining, presumably due to more careful use of gloves amon new hairdressers.

Permanent Wave Formulations

This section presents typical permanent wave formulations.

Formula 1, an amine bisulfite waving lotion, is buffered easily an is more stable than the sodium or ammonium bisulfite at an acid pH. Its inventors claim the penetration of the ethanolamine into the cortex is so much easier that no swelling agents are needed in the formulation. This is an improved formulation relative to the ammonium or sodium bisulfite wave lotions; however, it still remains slow Processing must be carried out under a heat drier. The product give soft body waves.

Formula 2 is a variation of a contemporary thioglycolate formula: "cold wave." This formula differs from the usual contemporary thiogly colate by containing mixtures of the ammonium and ethanolamine

The CHEMISTRY and MANUFACTURE of COSMETIC

522

Formula 1. Bisulfite Waving Composition (Hohenstein and Andrassy 1998)

Ingredients	Welght %
Glycerin or propylene glycol	5.00-10.00
• 7	2.00
Polysorbate 20	25.00
Ethanolamine sulfite, 60 %	3.20
Citric acid (qs to pH 6.1-6.50)	0.50
Fragrance (parfum)	qs 100.00
Water (aqua)	чз 100.00

Formula 2. Thioglycolate Waving Composition (Mathews et al. 1990)

Ingredients		Weight %
Ammonium thioglycolate		7.00
		2.00
Monoethanolamine thioglycolate		0.25
Chelating agent		6.00
Nonionic surfactant		0.25
Fragrance (parfum)		0.85
Aqua Ammonia (qs to pH 8.9 -9.2)	*	
2-Ethyl-1,3-Hexanediol		4.00
Water (aqua)		qs 100.00

the usual formula would contain 100% ammonium thioglycolate. Formula 2 would be considered a low-ammonia product, a category hair stylists recognize. The 2-ethyl-1,3-hexanediol is claimed to stimulate disulfide bond cleavage. Processing times can be reduced to about 5 minutes for an acceptable wave using hair-drier temperatures (50°C). Ordinarily, no heat is required; on chemically untreated hair, processing times can be as much as 20 to 25 minutes.

Formula 3 is a typical glyceryl thioglycolate perm formula. Excellent perming results are attained if the pH is less than 7, with or without heat. This is known to the stylists as a "thio" acid perm. To preserve the efficacy of the waving lotion, the active agent, glyceryl thioglycolate, must be mixed prior to usage. This prevents hydrolysis of the active agent but requires an additional bottle. This requirement of premixing hindered product marketing in Japan, Korea, China and elsewhere in Asia.

Formula 3. Glyceryl Thioglycolate Waving Composition (Mathews et al. 1989)

······································	
Ingredients	Weight %
Reformer	weight %
Glyceryl thioglycolate	100.00
Balancer	
Ammonia	0.34 - 0.85
Urea	1.00 - 15.00
Nonionic surfactant	
Fragrance (parfum)	1.00 - 6.00
" ,	0.10 - 0.50
Water (aqua)	qs 100.00
Reformer and Balancer are combined at time of use in prop 6.80-7.00.	ortions to provide a pH of

Formula 4 is known as a "thio free" formula. The waving lotion is considered to be mild since it leaves the hair soft and manageable with no hair damage. Excellent results are obtained on both chemically treated and non-treated hair. The curl results are typical of "thio acid" perms.

Formula 4. Cysteamine Waving Composition (Nandagiri et al. 1993)

Ingredients	Weight %
Cysteamine HCI	7.80 - 9.00
Ammonium hydroxide, to pH 7.8-8.8 Nonoxynol-15	qs
Fragrance (parlum)	0.90
Water (aqua)	0.30 qs 100.00

Formula 5 is also considered to be a "thio free" perm. Thiolactic acid is more hydrophobic than thioglycolic acid and hence its waving behavior is different than that of thioglycolate. Formula 5 is a low-ammonia, buffered perm with excellent results at pH 8.8-9.20.

Formula 6 has a much higher degree of safety in perming on diverse types of hair than ordinary waving lotions. The safety period of the

524

The CHEMISTRY and MANUFACTURE of COSMETICS

Formula 5 (Thiolactate Waving Composition) (Yasuhiro et al. 1996)

Ingredients	Weight %
Ammonium thiolactate	5.56
Monoethanolamine thiolactate	10.22
Moneoethanolamine, 99 %	1.21
Ammonium hydroxide 28 % -30 %	0.74
Ammonium bicarbonate	0.32
EDTA	0.05
Lauryl dimethylamine acetate betaine	0.50
Polyoxyethylene lauryl ether	2.00
Fragrance (parfum)	0.50
Phenoxypropanol	0.50
Amodimethicone	1.00
Water (aqua)	qs 100.00

Formula 6. Thioglycolate/Dithiodiglycolate Ester or Salt Waving Composition (Klemm, Edman et al. 1981)

Ingredients	Weight %
Thioglycolate ester or salt	10.00 - 30.50
Dithiodiglycolate ester or salt	0.00 - 15.00
Polyhydroxyl compound	0.00 - 15.00
Salt	0.00 - 3.00
Water (aqua)	qs 100.00
	•
Alkanolamines, ammonia or carbonates of ammor	nia may be used for pH adjustments

waying lotions in contact with hair increases with desirable end results the perming especially on chemically or mechanically damaged hair.

In Formula 7, the active agent is a dithiol low molecular weight polymers. Excellent perming results are obtained on chemically damaged or porous hair. The corresponding disulfides of this dithiol agent appear to have a repair effect on chemically damaged hair. The active agent must be premixed immediately prior to usage to preserve efficiency of the product.

ERMANENT WAVING OF HAIR

Formula 7. Polyethylene Glycol Dimercapto Acetate (Savaides and Saice 1994)

Ingredients	Weight %
Polyethylene glycol dimercapto-acetate (mw 540)	15.00-30.00
Nonionic surfactant	1,50-2.00
Fragrance (parfum)	0.50
Ammonia, to pH 8.50–8.80	qs.
Water (aqua)	qs 100.00

Formulation of Neutralizers

The active ingredients in the neutralizer formulations are oxidizing agents. These oxidizing agents may include hydrogen peroxide, perborates, ammonium persulfate and sodium or potassium bromate. The most widely used oxidizing agent in the neutralizers is hydrogen peroxide. Hydrogen peroxide is highly active at neutral and alkaline medium than at acid medium. However the stability of the hydrogen peroxide is maximized at pH 3-4. The decomposition of hydrogen peroxide is minimized at an acid medium. Stabilizers such as phosphoric, tartaric and or citric acid may also be added into the formula. Water quality is critical and it must be absolutely free of any iron traces.

Formula 8 is an emulsified neutralizer formula from Heilingotter (1975).

Formula 8. Neutralizer Formulation

Ingredients	Weight %
A. Cetyl alcohol	2.50
PEG-100 stearate	2.50
B. Water (aqua)	86.43
C. Hydrogen peroxide, 35 %	8.57
D. Phosphoric acid, 10%, to pH 3.5-4.0	qs

Procedure: Heat A to 70°C; mix thoroughly. Heat B to 72°C. Add B to A slowly, with mechanical mixing. Allow the emulsion to cool at room temperature with slow mixing. Add C slowly and adjust pH to 3.5-4.0 by adding D.

526

The CHEMISTRY and MANUFACTURE of COSMETICS

THIS PAGE BLANK (USPTQ)

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:
☐ BLACK BORDERS
IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
☐ GRAY SCALE DOCUMENTS
LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
OTHER:

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

THIS PAGE BLANK (USPTO)